MULTI-SCALE APPROACH TO INVESTIGATE THE TENSILE AND FRACTURE BEHAVIOR OF NANO COMPOSITE MATERIALS

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Narrative Description of the Program:

This program is concerned with the effects of nano size particles on the tensile and fracture behavior of particulate composite materials. The program's basic approach involves a blend of experimental and analytical studies. In general, mechanisms and mechanics involved in the damage process and cohesive fracture are emphasized. Special issues that will be addressed are: (1) To what extent and by what mechanism the nano size particles affect damage initiation and evolution processes, deformation process, and crack growth behavior? (2) What are the deformation and failure mechanisms on the meso and the macro levels? (3) How do the nano size particles affect the characteristics of the interphase between the particle and binder? And (4) what is role of the interphase properties in damage initiation and evolution processes?

The objectives of the proposed research are to (1) obtain a fundamental understanding of the effects of nano size aluminum particles on the constitutive and crack growth behavior of particulate composite materials, (2) investigate the effects of aluminum particle size on deformation mechanism, damage process, hysteresis, and fracture strength under a constant strain rate condition, (3) determine the role of the interphase in damage initiation and evolution processes, (4) determine the deformation and failure mechanisms on meso and macro scales, (5) develop a microstructural and statistical based technology to evaluate the inherent material quality ,and (6) provide guidance for developing high strength solid propellants containing nano size particles.

In this program, uniaxial tensile tests will be conducted on specimens with and without pre-crack to determine the constitutive and fracture behavior under a constant strain rate condition. Digital Image Correlation techniques will be used to determine the strain field on the surface of the specimen. Ultrasonic and high-resolution x-ray radiograph techniques will be used to determine the damage initiation and evolution processes in the material. The correlation of the damage state and the constitutive and fracture behavior will be determined. Statistical mechanics and fracture mechanics will be used to determine the inherent quality of the material. Multi-scale numerical modeling techniques will be used to simulate the damage and fracture processes in the material. In addition, damage mechanics, on the meso- scale, and fracture mechanics, on the macro-scale, will be used to simulate the crack growth behavior.

Detailed Technical Approach for FY04:

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13. SUPPLEMENTARY NOTES

14. ABSTRACT
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15. SUBJECT TERMS

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In FY 04, there are three major tasks: Task 1 – Meso and Macro Scale Strain Measurements, Task 2 – Multi-Scale Modeling on constitutive and crack growth behavior, and Task 3 – Cumulative Damage Analysis

Task 1 – Meso and Macro Scale Strain Measurements:

In FY 03, the deformation and failure mechanisms in two matrix materials [Solithane 113, Tetrahydrofuran-Polyethylene Glycol (TPEG)]) and a composite material (TPEG with 10% by weight of 6 micron aluminum particles, and it is denoted as Composite 1) were investigated. In FY 04, uniaxial specimens, which were made of TPEG with 10% by weight of 0.2 micron aluminum particles and denoted as Composite 2, were tested under a constant strain rate condition. In addition, crack propagation tests as well as cyclic loading and constant strain tests were conducted on specimens made of the two composite materials. Tests were performed in a Hitachi scanning electron microscope (model S-2460N), which was equipped with a displacement controlled loading device. The crosshead of the loading device can travel continuously. The Computer Aided Speckle Interferometry technique was used to analyze the test data

Experimental findings revealed that the strain distributions varied with the size of the area, A, in which the data were analyzed. The area, $A_{u,}$ in which the strain distributions were relatively uniform, varied with the three materials investigated. These experimental observations reveal that there exists a length scale below which the material's microstructure has a significant effect on the strain distributions. In other words, a representative area, which is defined as an area in which the material's microstructure has no significant effect on the strain distribution, exists for the materials investigated in this study. It is interesting to point out that for the three materials investigated, the normal strain distributions measured in a small area of 0.002 mm^2 at 1500X are highly nonuniform and both tensile and compressive strain fields exist. In addition, for the composite material, positive transverse strains were developed at certain locations in the materials. The triaxial tensile strain fields are the potential failure locations in the materials.

In addition to determining the strain fields, the damage mechanisms near the crack tip were also investigated. Although the deformation mechanisms, large displacement and ligament formation, near the crack tip of the three materials were similar, the damage mechanisms near the crack tip depended on the material. For TPEG, no through-thickness voids were formed near the crack tip. Instead, microcracks were formed in the ligaments (Fig.1). The growth of the microcrack resulted in the fracture of the ligament. For the composite materials, voids were formed around the AL particles (Fig.1). As the applied strain was increased, the void elongated, resulting in a higher toughness of the material and slow crack growth rate.

Plots of crack length versus of time for the two composite materials are shown in Fig. 2. Plots of relaxation modulus versus time and available elastic energy versus number of cycle are shown in Figs. 3 and 4, respectively. From Fig. 2, it is noted that the crack

grows faster in Composite 2 than Composite 1. Experimental data show that the critical load for the onset of crack growth for Composite 2 is higher than that for Composite 1. The higher crack growth rate observed in Composite 2 is mainly due the increase in the relaxation modulus (Fig.3) and the available elastic energy (Fig.4) for crack propagation. From Fig.3, we noted that the aluminum particles change the relaxation behavior of the materials. Therefore, if we compare the material response of the three materials at the same strain rate we would find that the composite materials are stiffer than TPEG and Composite 2 is stronger than Composite 1.

Task 2 – Multi-Scale Modeling on Damage Initiation and Evolution

In this task, the constitutive and the crack growth behavior in a solid propellant were simulated using a multi-scale technique. In this approach, the macro-level (i.e. composite specimen level) and the micro-level (i.e. the particle and matrix material level) are interconnected in analysis in a staggered way.

In simulating the constitutive behavior of the material, the specimen of size 24 mm. by 24 mm was meshed into sub-domains of the size of the characteristic area, which was determined in FY03, and it had a size of 2 mm. by 2 mm. In each sub-domain, the material was divided into 16 elements with each element having a different volume fraction of particles (VFP). However, the average VFP in the sub-domains were the same, which was equal to 0.65 with a standard deviation of 0.1. The variations in VFP were determined using normally distributed random number generation. Once normally distributed random numbers were generated for each sub-domain, their values were scaled properly to maintain the average volume fraction of particle over each sub-domain.

Figure 5 shows the simulated stress-strain curves for a specimen with uniformly distributed VFP and for specimens with different characteristic areas in which the VFP of each elements are different. It is seen that the stiffness is almost the same among different specimens regardless of the size of the characteristic area. Even the uniform specimen, which has no variation of particle density from element to element, has almost the same stiffness as the non-uniform specimens. However, the failure strengths were lower for the specimens with non-uniform VFP when compared with the uniform VFP case. On the other hand, different characteristic sizes of the sub-domains have the same failure strength.

The next study was focused on the simulation of crack growth behavior of a centrally-cracked specimen. In the analysis, a global analysis was conducted first to determine the deformation in the specimen. From this analysis, the deformation around the crack tip, determined from the global analysis, was used for the local analysis, which had a vary fined mesh around the crack tip. The global analysis model had 144 elements that had the same VFP for every element while the local analysis model had six elements, each of which was sub-divided into 256 elements. The VFP of the 256 elements varied from element to element with the average VFP the same as that used in the global analysis. In

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addition, for a comparison purpose, a uniformly distributed VFP case including the local elements was conducted.

In the analysis, the size of the saturated damage zone was considered as the incremental crack length. For the cases of random distribution of particles, the analysis was repeated five times with normal random generation for statistical analysis. The results of the analysis, shown by plotting the mean crack length with scatter bar against the applied load, are shown in Fig.6. The results showed that by comparing with the uniformly distributed VFP case, the randomly distributed VFP case resulted in crack growth under a smaller applied strain and a longer crack length for a given applied load.

In addition to conducting multi-scale analysis to predict the constitutive and crack growth behavior, we also developed a technique, based on a combination of the boundary element method and the homogenization method, to investigate the effect of particle size and arrangement on the material property of particulate composites. The results of the modeling analyses reveal that, for perfect bonding between the particle and the matrix, particle arrangement (regular and random) and particle size have no effect on the predicted effective Young's modulus and Poisson's ration as long as the volume fraction of particle is the same. For both Composites 1and 2, the predicted Young's modulus and Poisson's ratio are 0.51 MPa and 0.4999, respectively. However, experimental results show that the Young's modulus for Composite 1 and Composite 2 are 0.53 MPa and 0.66 MPa, respectively. It is seen that the predicted Young's modulus for Composite 1 compares well with the measured value. However, the predicted and the measured values for Composite 2 differ by 20%. Currently, we are conducting additional studies in order to explain the discrepancy between the predicted and measured Young's modulus for Composite 2.

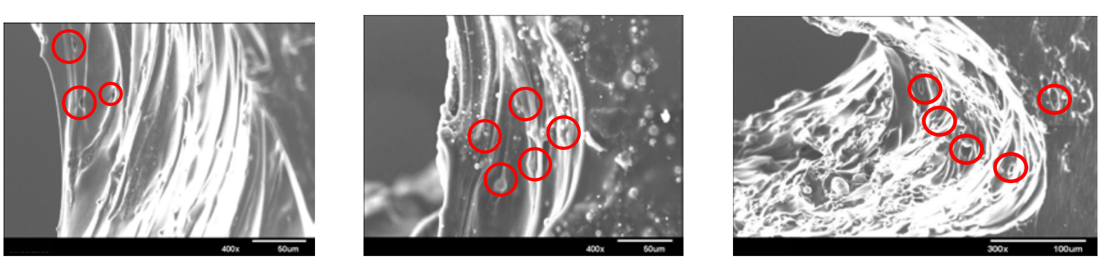
Task 3 – Cumulative Damage Analysis

The microstructure change and the crack formation in a solid propellant subjected to an incremental strain loading condition were investigated using real-time x-ray techniques. During the test, Lockheed-Martin Research Laboratory's High-Resolution Digital X-Ray System was used to investigate the characteristics of the damage initiation and evolution processes. An Instron table model tensile testing machine, which is placed between the x-ray radiation source and the x-ray camera, was used to strain the specimen under an incremental strain condition. In addition, a CCD camera was placed on the top of the X-ray radiation source, and it was used to monitor the deformation on the surface of the specimen during the test. The recorded x-ray data were processed to create a visual indication of the energy absorbed in the material. The deformation data recorded on the tape were used to determine the strain fields using an imaging correlation technique. The load and the displacement data recorded from the Instron testing machine were used to calculate the stress and the strain. This unique testing set up can be used to collect x-ray data and mechanical data simultaneously during the test. By processing the experimental data, we can obtain information on micro-structure evolution, damage process, damage state, stress-strain constitutive behavior, and strain fields on the surface of the specimen

X-ray data reveal that, prior to stretching the specimen, the color of the specimen is relative uniform, indicating that the material's microstructure is relatively uniform. As the applied strain level is increased, the color varies along the length of the specimen. The non-uniform distribution of the color is due to the change of the microstructure of the material. It is interesting and important to point out that, at a critical applied strain level, a crack is formed in the weak region, and it doesn't propagate. As the applied strain level is increased, the number of the non-propagating cracks increases. Finally, two non-propagating cracks coalesce, resulting in a long crack which propagates and leads to the fracture of the specimen. Experimental findings reveal that the degree of inhomogeneity of the material's microstructure and the number of non-propagating crack increases as the applied strain is increased. Also, the strain distribution is highly non-uniform when the applied strain is high.

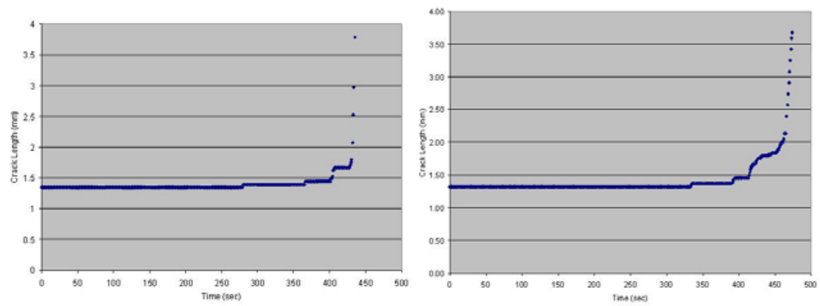
In addition to investigating micro-structural change, damage initiation and evolution processes, and crack growth behavior a technique was developed, based on x-ray data, to predict volume dilatation, ΔV/Vo, as a function of applied strain. The volume dilatation is a physical damage parameter and is used to develop a nonlinear constitutive model, which is incorporated in a computer code and used to predict the non-linear constitutive and crack growth behavior in solid propellants with good accuracy. The volume dilatations predicted from x-ray data and measured by dilatometer are shown in Fig.7. It can be seen that a good correlation exists between the predicted and the measured dilatations. This experimental finding gives us confidence in using the x-ray technique to predict volume dilatation and the critical x-ray intensity for the onset of crack growth, which is a subject of a current study.

Currently, we are conducting a detailed statistical analysis to relate the statistical parameters to damage evolution process and crack formation. In addition, crack propagation tests will be conducted to see whether or not we can use the x-ray technique to predict the onset of crack growth. Also, numerical modeling techniques will be used to investigate the effect of micro-structure on the non-propagating crack.



(a) TPEG (b) Composite 2 (c) Composite 1

Fig. 1 Local Behavior at Crack Tip



(a) Composite 2 (b) Composite 1

Fig. 2 Crack Length versus Time Curves.

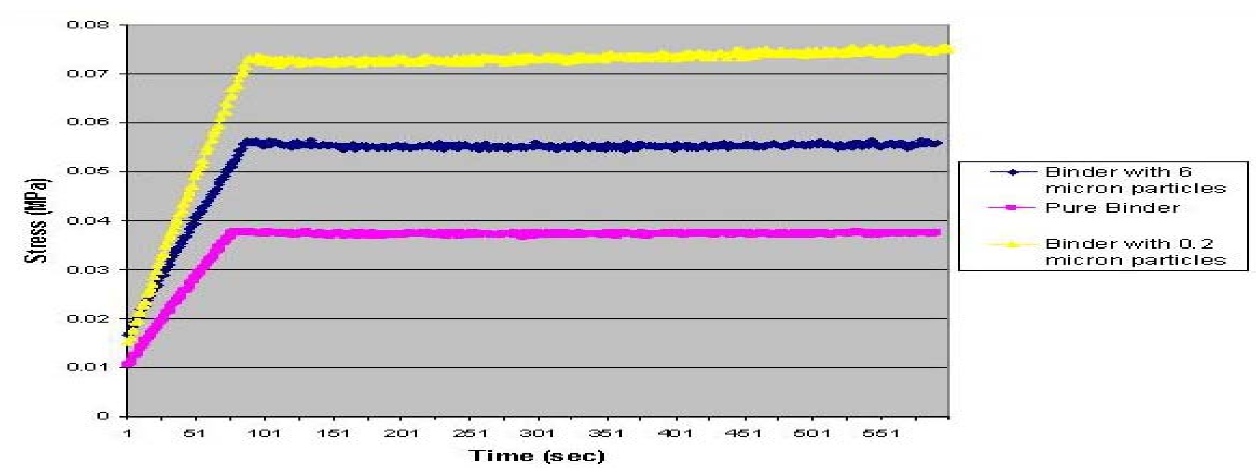


Fig.3 Stress Relaxation Curves.

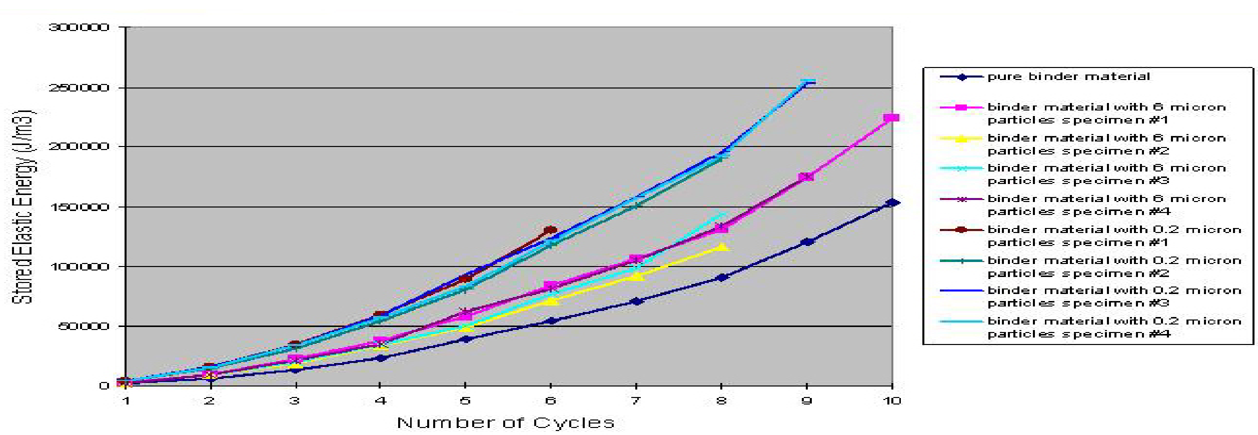


Fig. 4 Stored Elastic Energy Curves.

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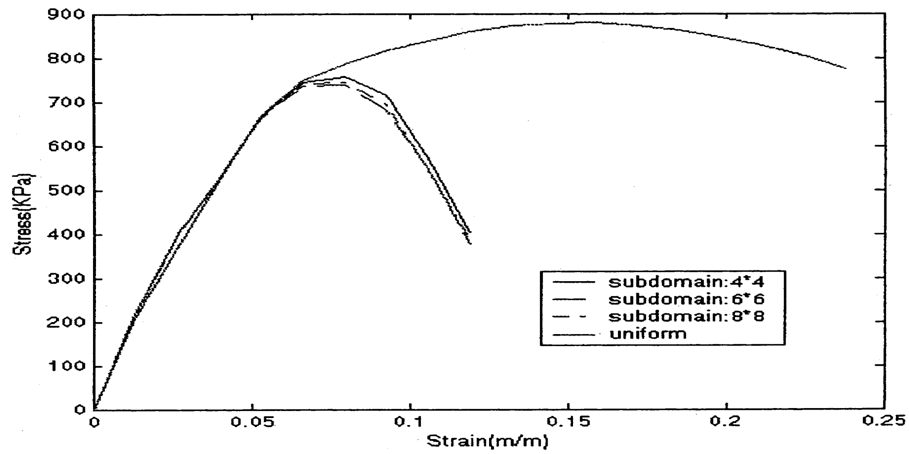

Fig. 5 Stress versus Strain Curves.

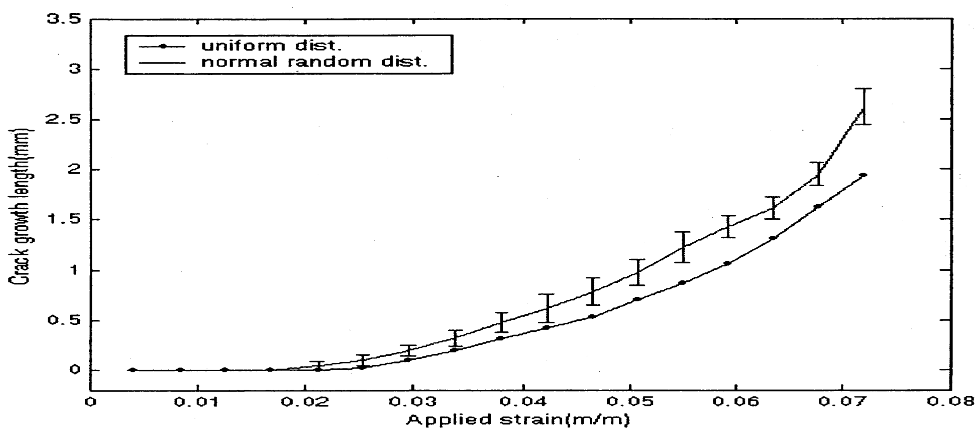

Fig. 6 Crack Length versus Applied Strain Curves.

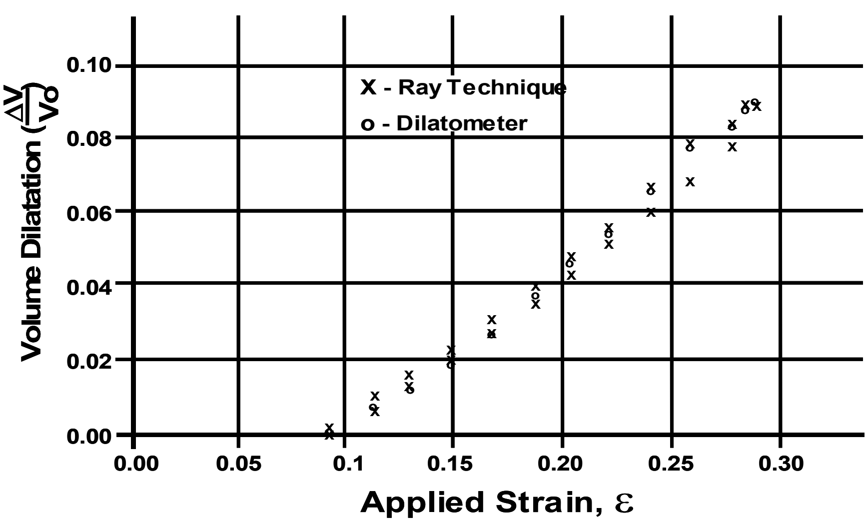

Fig. 7 Volume Dilatation versus Applied Strain Curves.

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Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials.

AFOSR Program Review

18 - 20 August 2004

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AFRL/PRSM

Edwards AFB CA.

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Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials.

- **Objectives:**
  - Obtain a fundamental understanding of the tensile and fracture behavior of nano composite materials.
  - Develop a microstructure and statistical based technology to evaluate the inherent material quality.
- **State of the Art:**
  - Uniaxial tensile and combustion characteristics tests were conducted.
  - Fracture behavior not studied.
- **Approaches:**
  - Multi-scale experimental, analytical, and numerical modeling analyses
  - Damage mechanics, experimental mechanics, fracture mechanics, and statistical mechanics
- **Applications:**
  - Strategic and tactical missile systems.

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Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials.

- **Past Year Accomplishments:**
  - Conducted constant strain rate and cyclic loading tests on a composite material (TPEG and 10% by weight of 0.2 micron AL particles).
  - Investigated the deformation and failure mechanisms.
  - Conducted X-ray tests to determine micro structural evolution, damage process, and crack formation in a solid propellant
  - Conducted numerical modeling analyses to (1) investigate the effect of microstructure on the constitutive behavior of a solid propellant and (2) predict the effective material properties of the solid propellant.
- **Research Payoff:**
  - Provide a fundamental understanding of the role of nano size particles on the deformation and damage processes as well as crack growth behavior.
  - Provide guidance for developing high strength nano composite materials.
- **Related Research Program:**
  - EPFC Program (AFRL/PRSP)

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Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials.

Uniqueness of Research:

- Unique Material (dual function and highly filled multi-size particles material).
- Account for microstructural effect on tensile and crack growth behavior.
- Account for local time-dependent behavior in crack growth simulation.
- Multi-scale microstructure controlling factors for damage and crack growth.
- Bridge the gap between meso and macro analyses.

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Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials.

Success Story:

- There is no success story yet, because this four-year program just started in FY 03.

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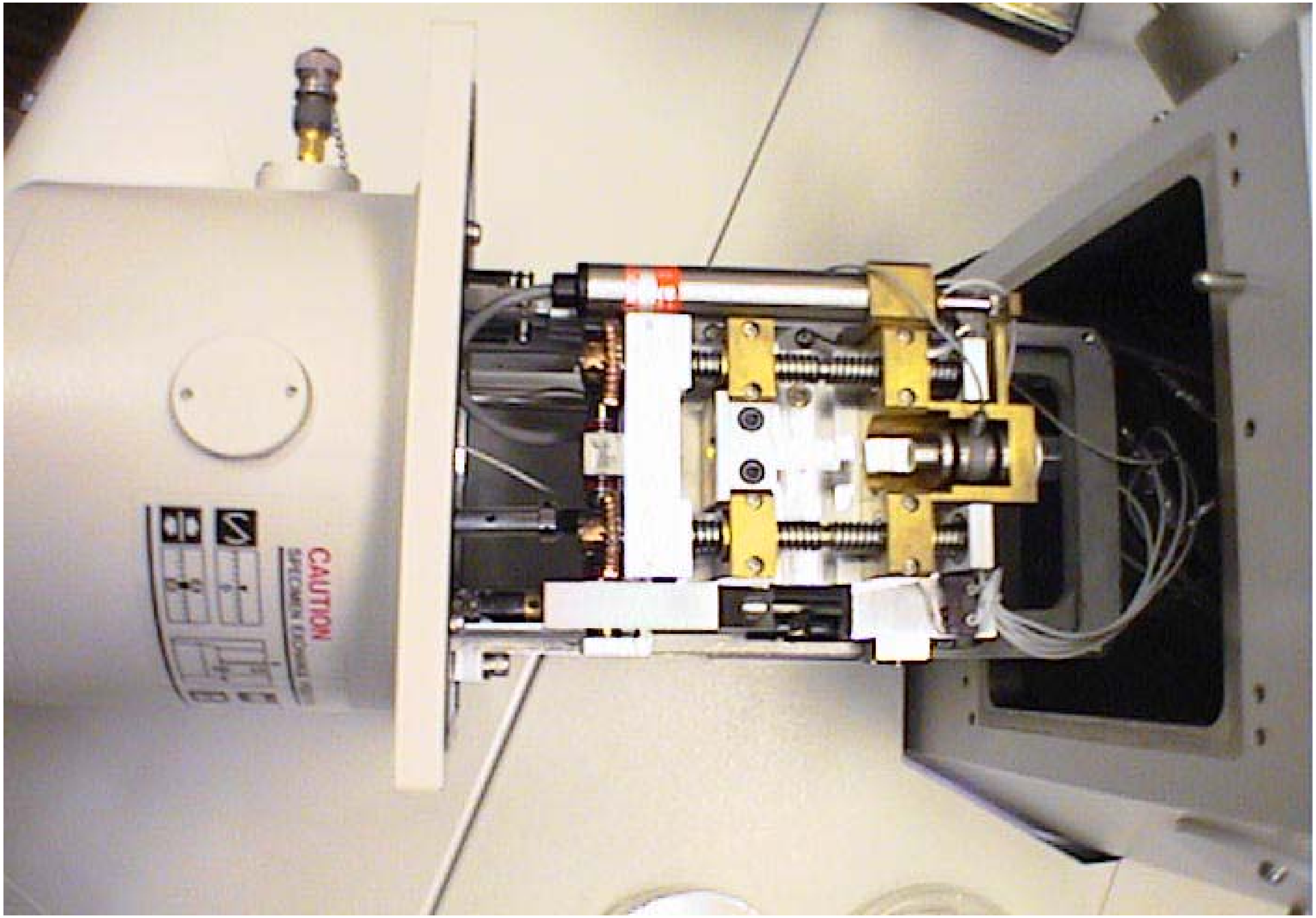
Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials.

Applications:

- **The developed techniques can be used to formulate high performance solid propellants for future strategic and tactical missile systems.**

Testing Set-Up



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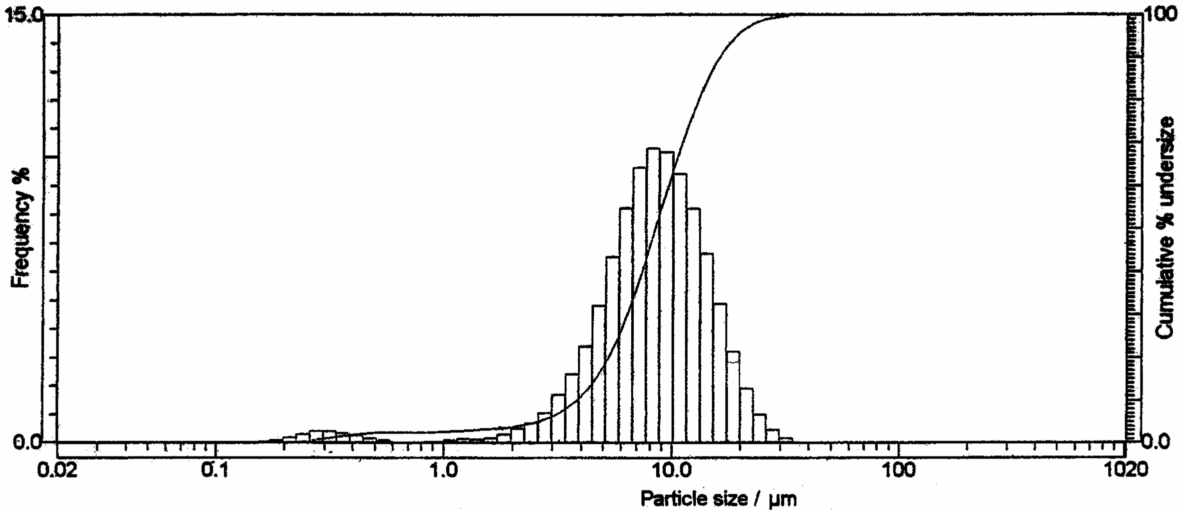
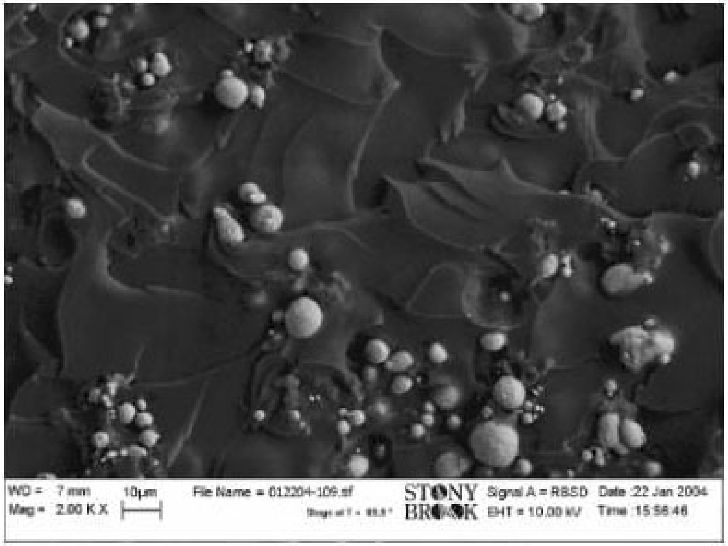
SEM Pictures of Fracture Surfaces of Binder Material with 6 micron Aluminum Particles





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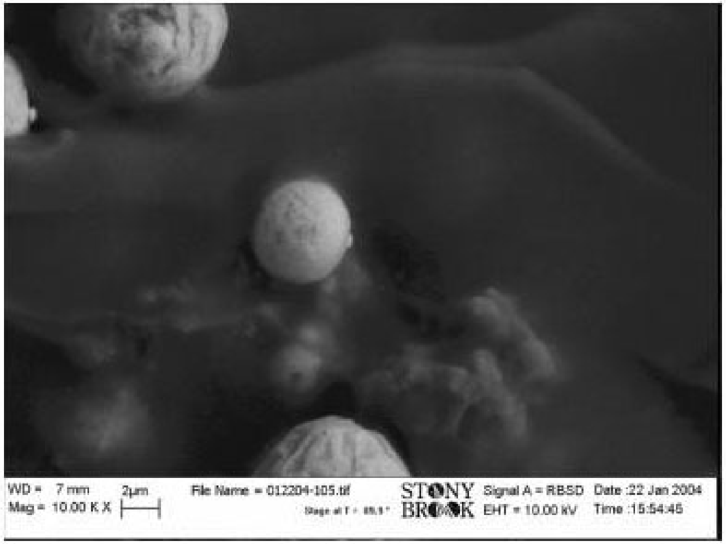

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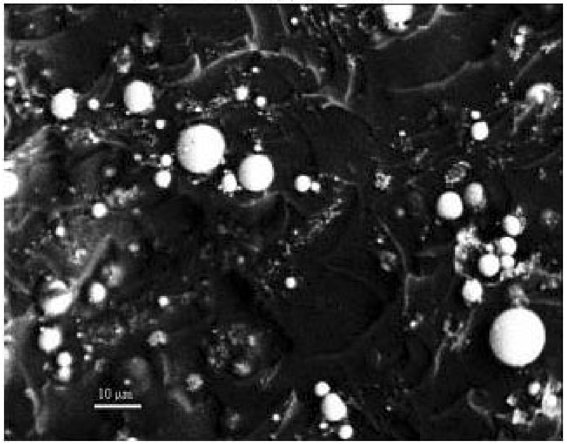
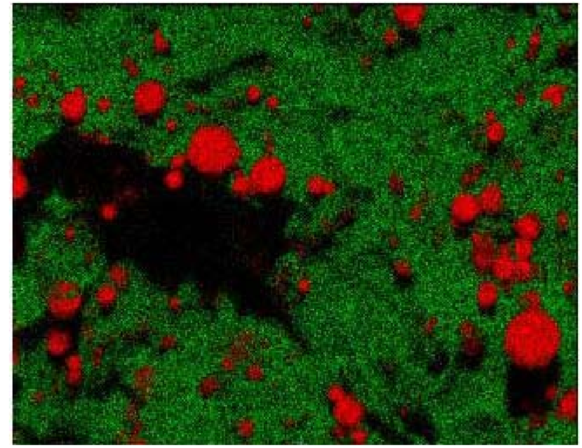
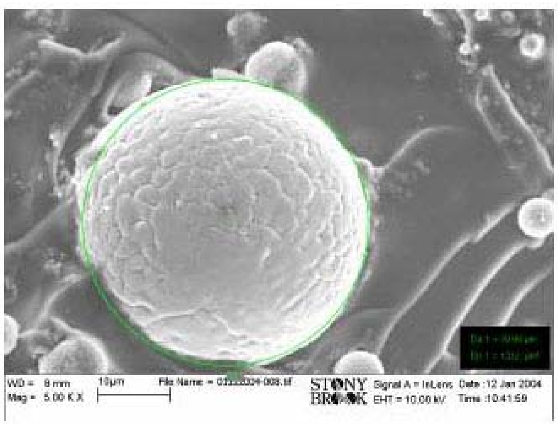
SEM Pictures of Fracture Surfaces of Binder Material with 0.2 μm Aluminum Particles



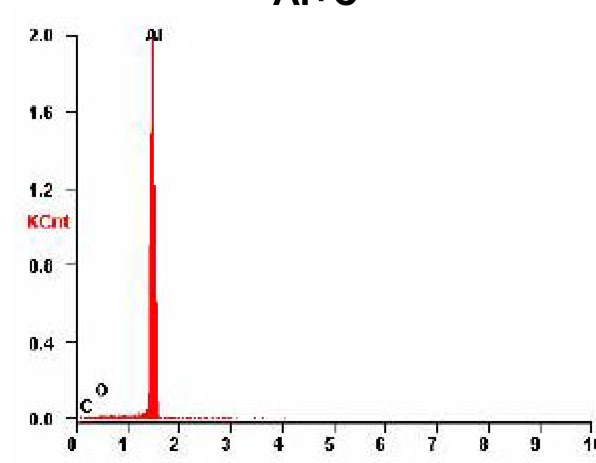
5000x



Al+C



Binder Material with 0.2μm Aluminum Particles (2000x)



Corresponding EDAX Spectrum

The interfacial strength between the AL particle and the binder is weak.

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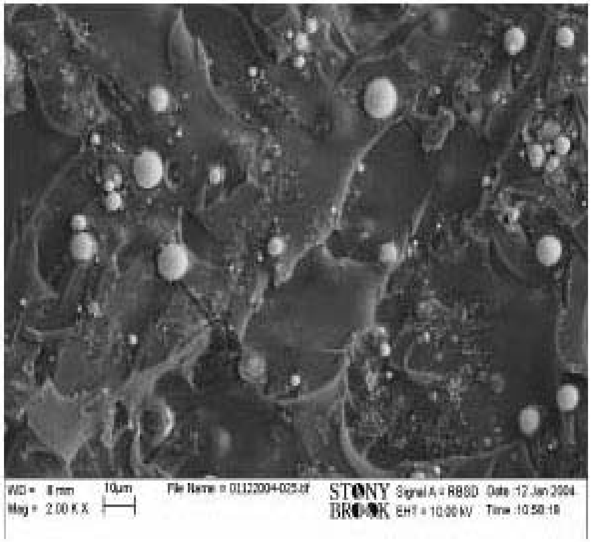
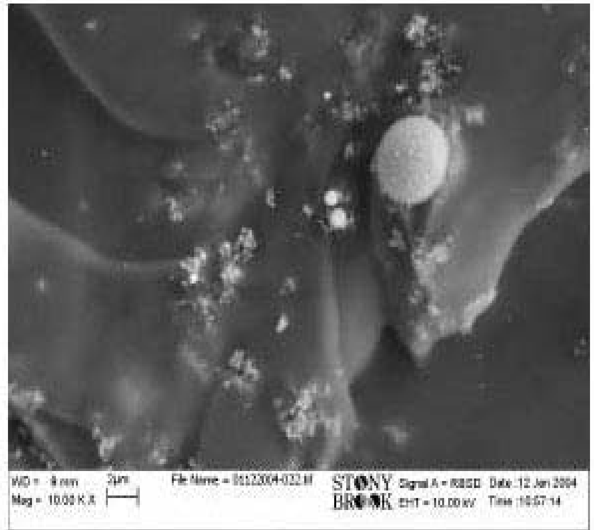
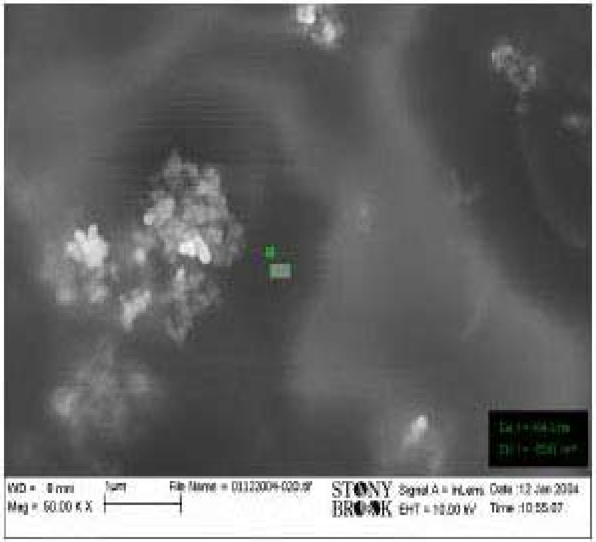
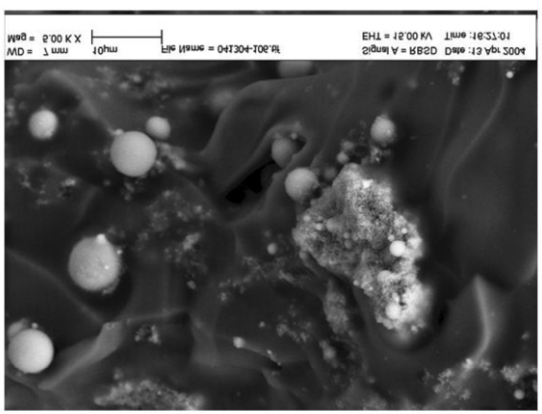
SEM Pictures of Fracture Surfaces of Binder Material with 0.2 micron Aluminum Particles

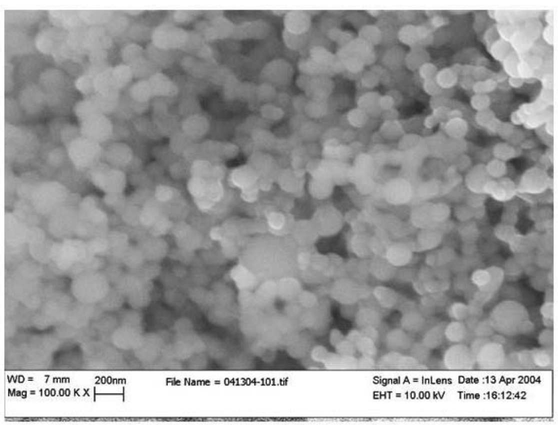


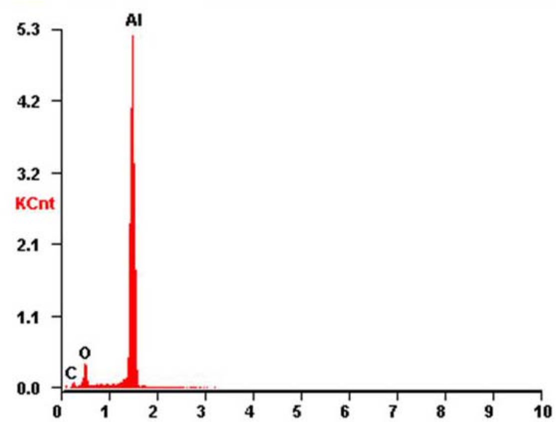




2,000x **20,000x** **50,000x**







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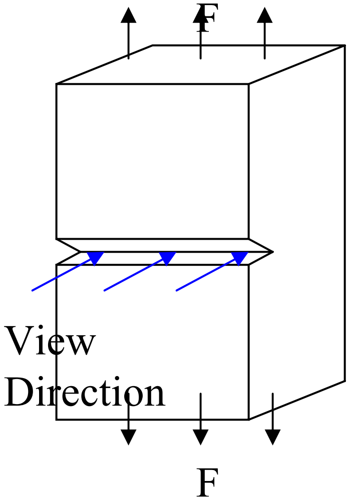
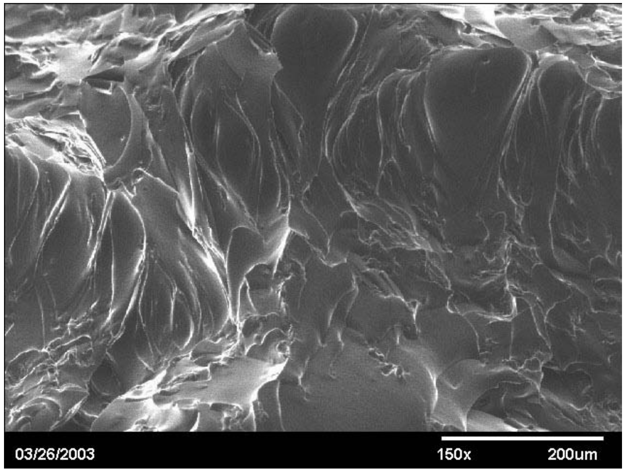
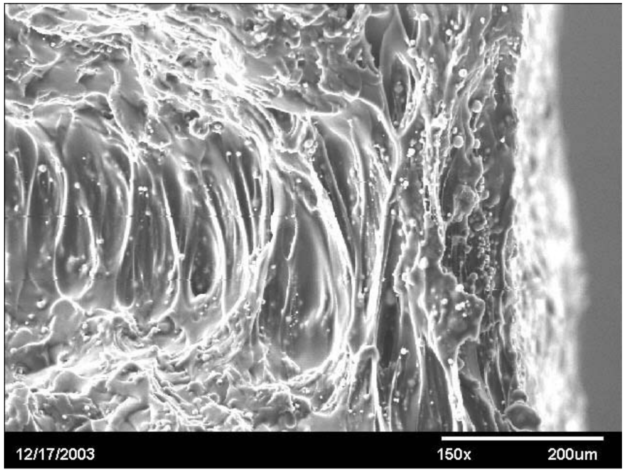
Local Deformation and Failure Mechanisms at Crack Tip (Top View)

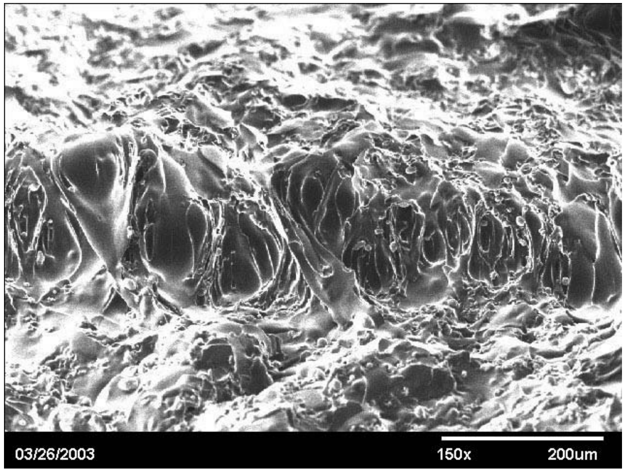




Pure Binder



Pure Binder with 0.2 micron AL Particles



Pure Binder with 6 micron AL Particles

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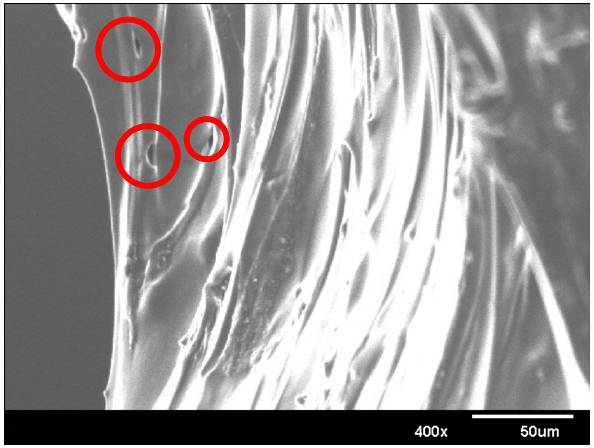
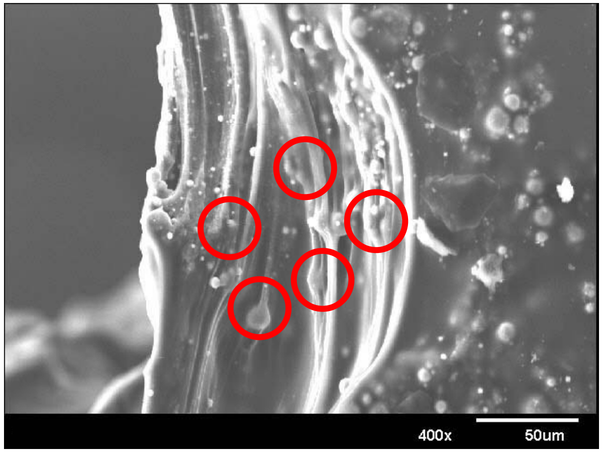
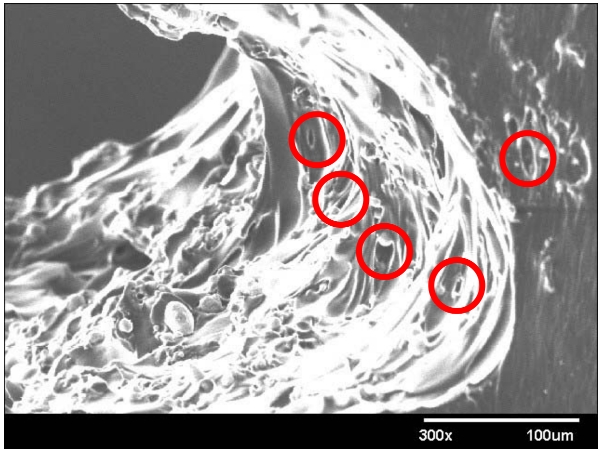
Local Deformation and Failure Mechanisms at Crack Tip (Side View)



Pure Binder



Pure Binder with 0.2 micron AL Particles



Pure Binder with 6 micron AL Particles

- **For the three materials studied, the local deformation mechanisms near the crack tip are similar**
- **The failure mechanisms in the pure binder material and the AL particles reinforced binder are different**

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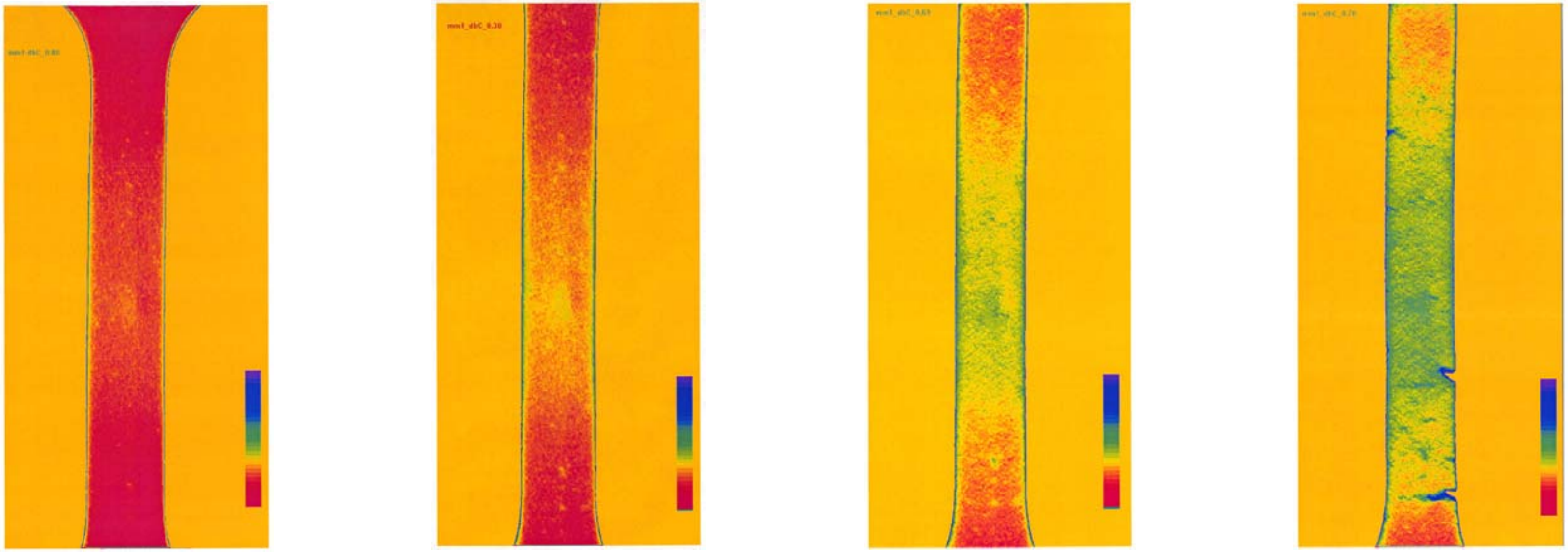
Micro-structure Evolution and Formation of Non-Propagation Cracks



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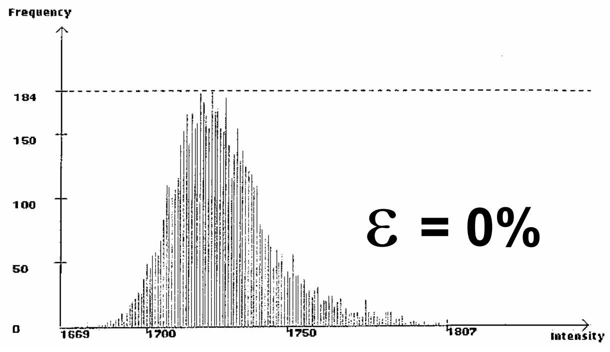
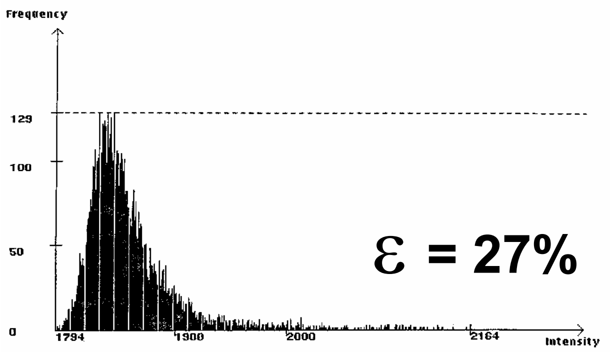
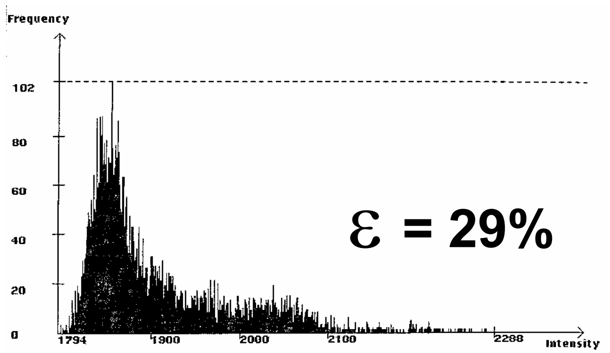
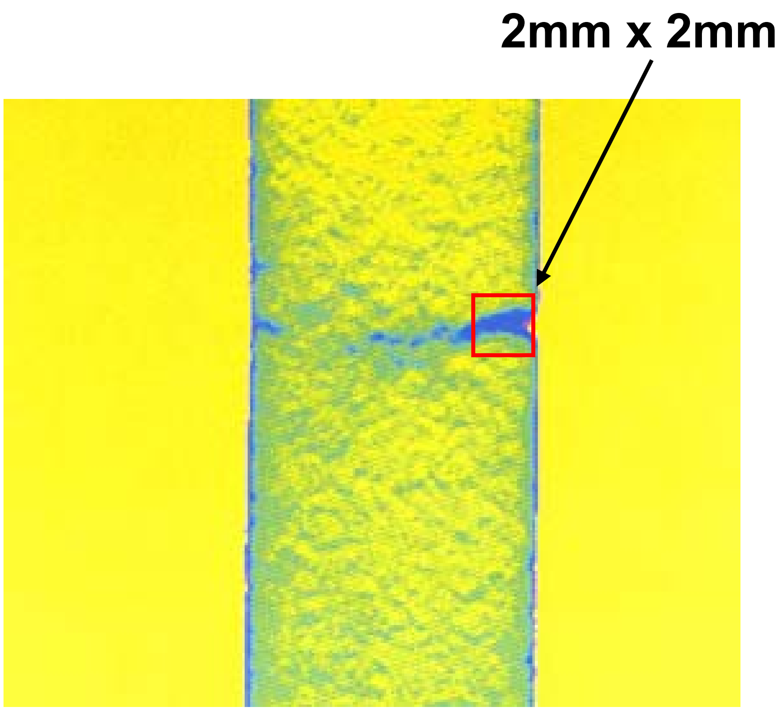
Histogram of X-Ray Intensity, or Damage Intensity, as a Function of Applied Strain

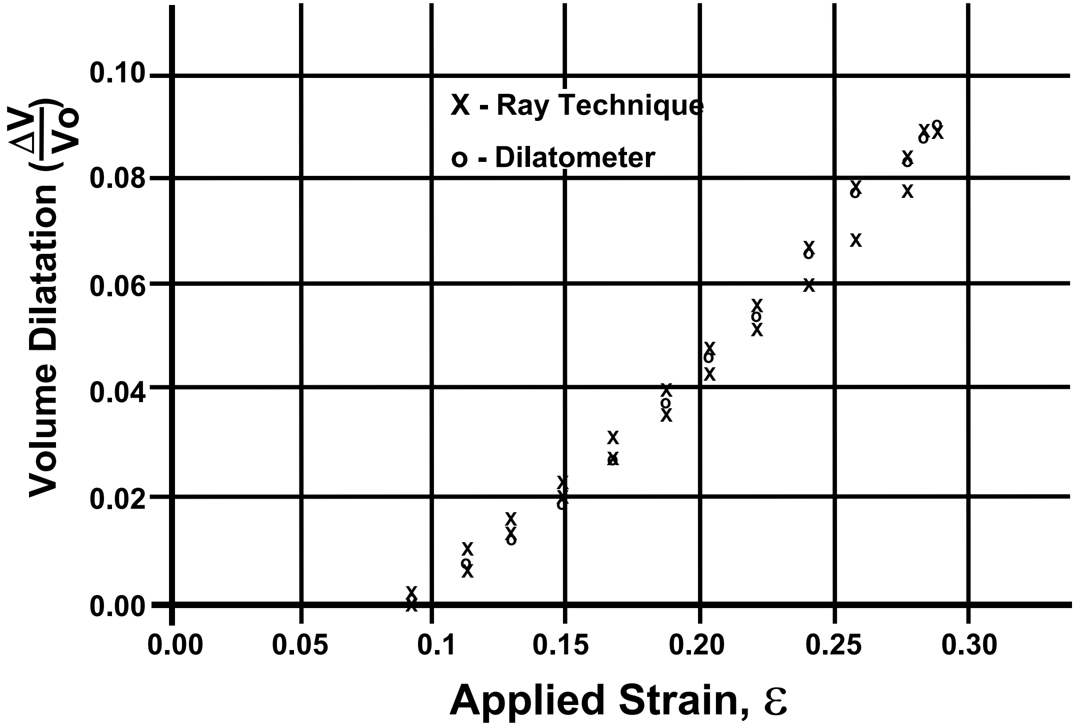








A Good Correlation Exists Between the Dilatations Measured by X-ray Techniques and Dilatometer



- **Provide a technique to nondestructively measure volume dilatation, which is a physical damage parameter.**
- **The volume dilatation can be used to model the non-linear behavior of solid propellants and to predict the critical J-integral value for the onset of crack growth.**

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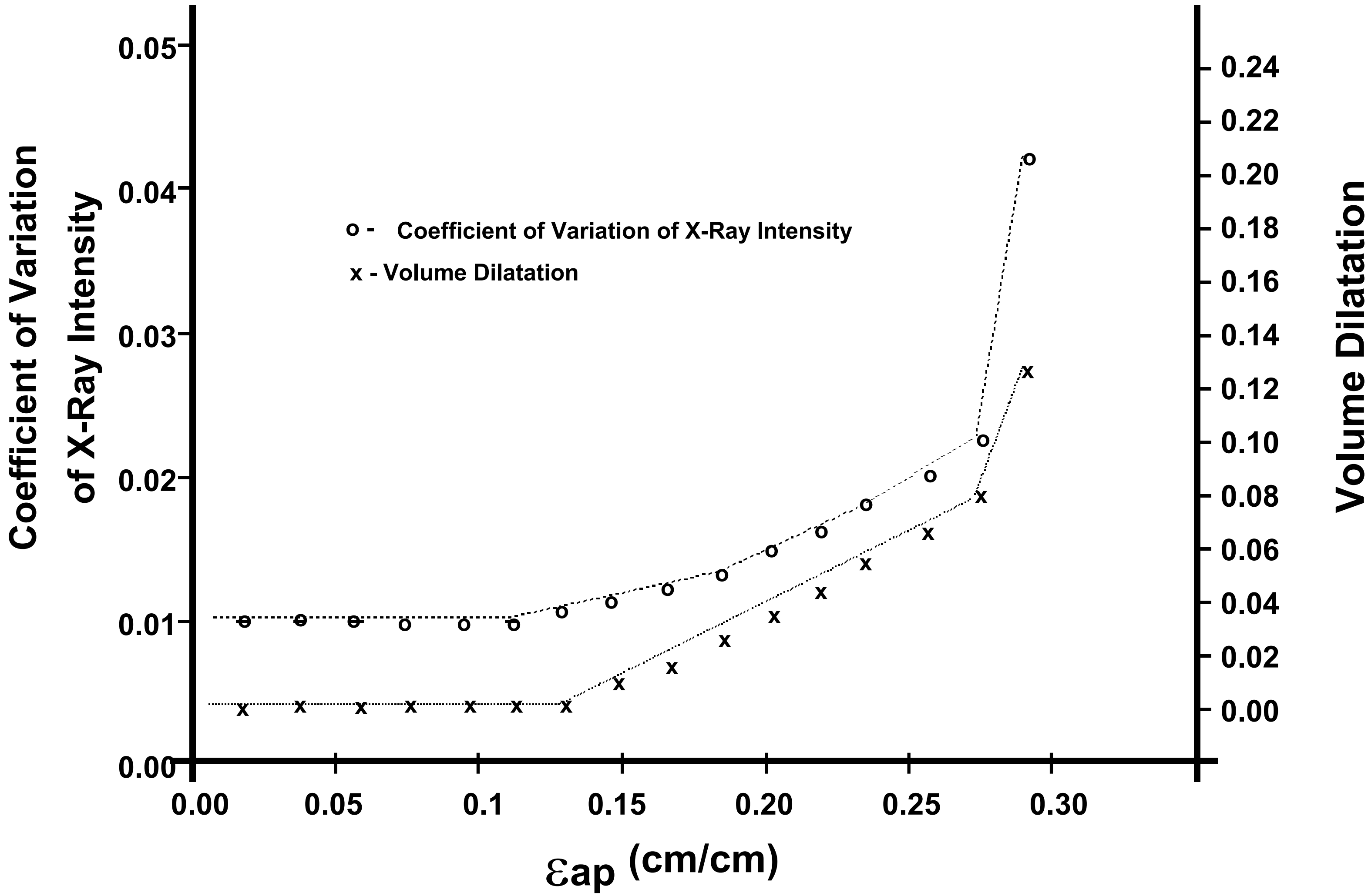
X-ray Technique Is a Promising Method to Monitor Damage Evolution in Solid Propellants



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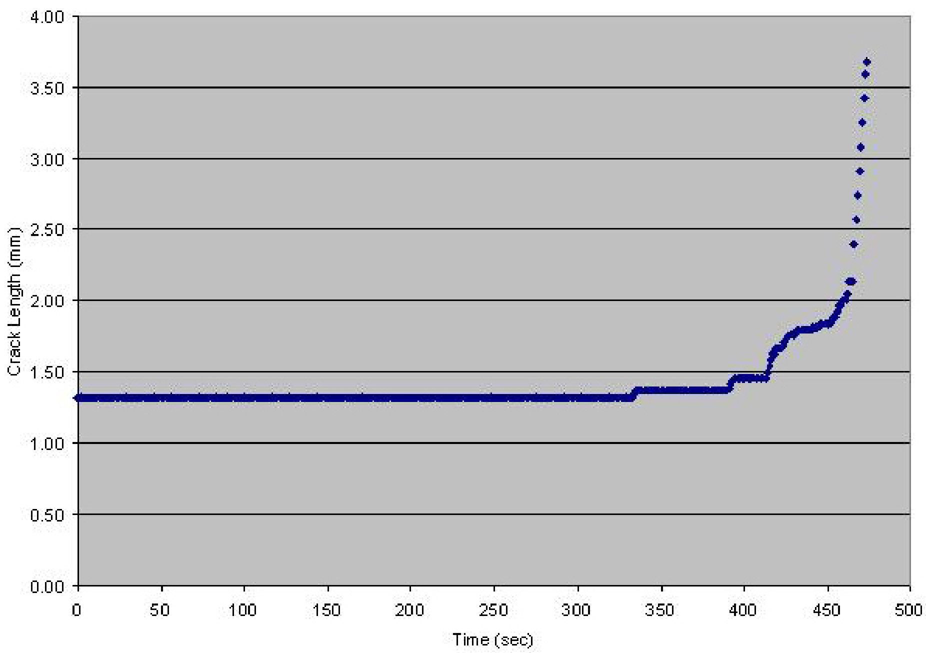
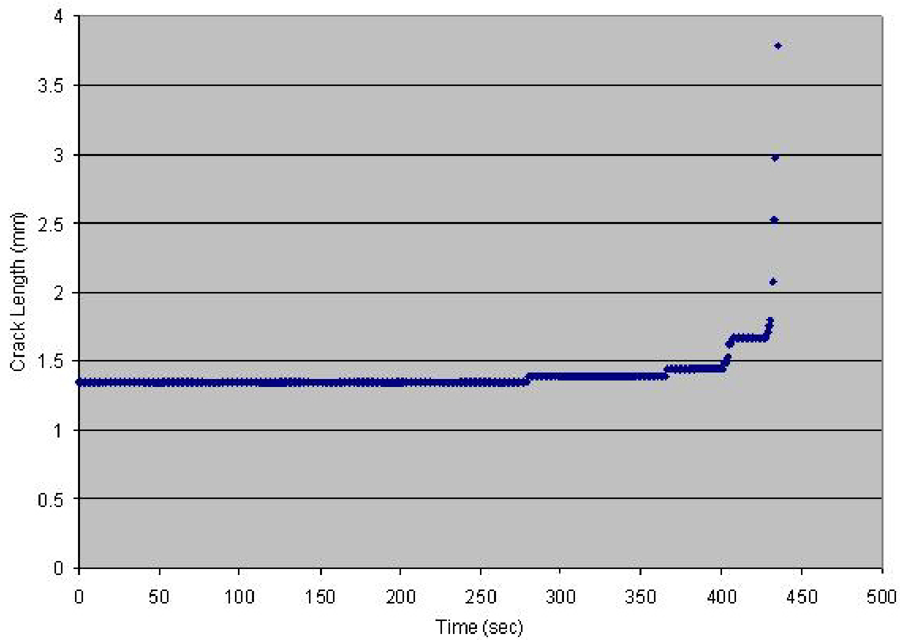
Crack Growth Curves



Pure Binder with 6 Micron AL Particles



Pure Binder with 0.2 Micron AL Particles

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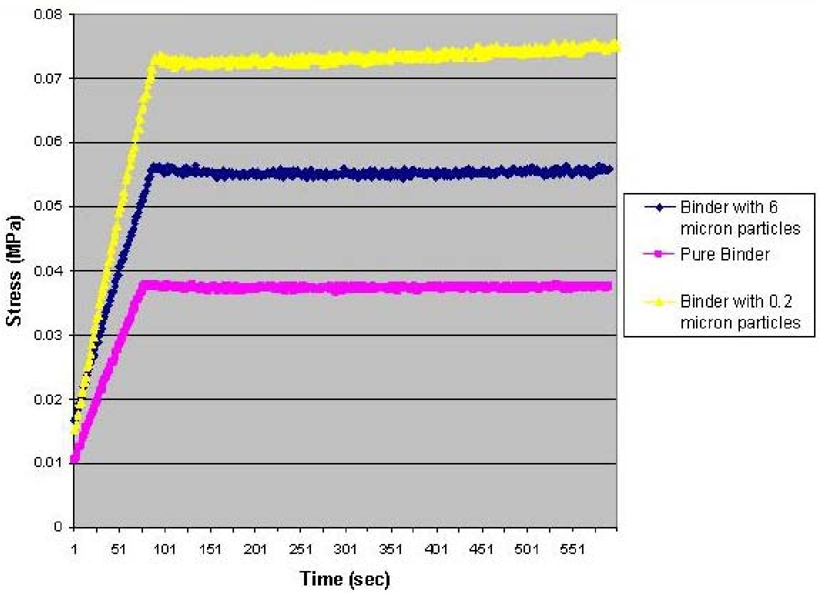
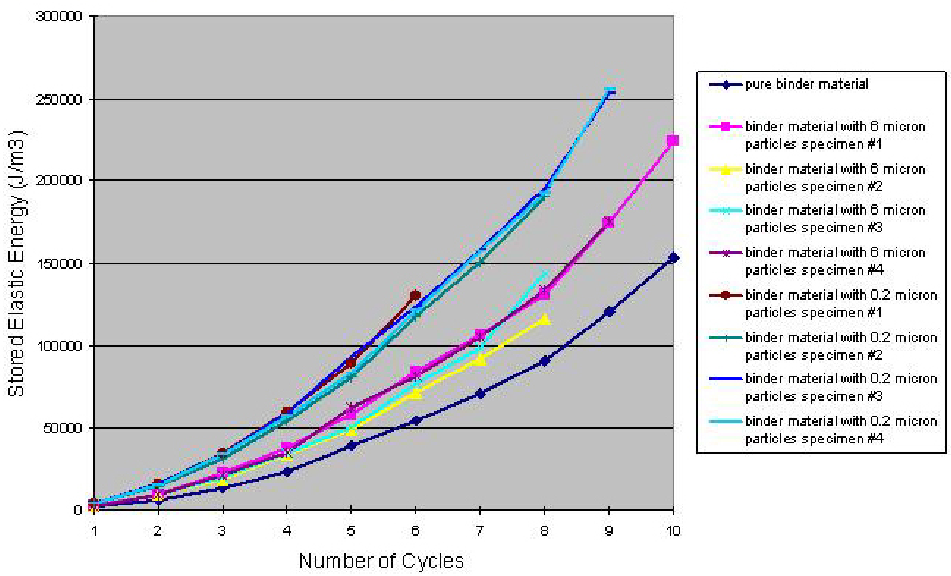
Stored Elastic Energy and Stress Relaxation Data





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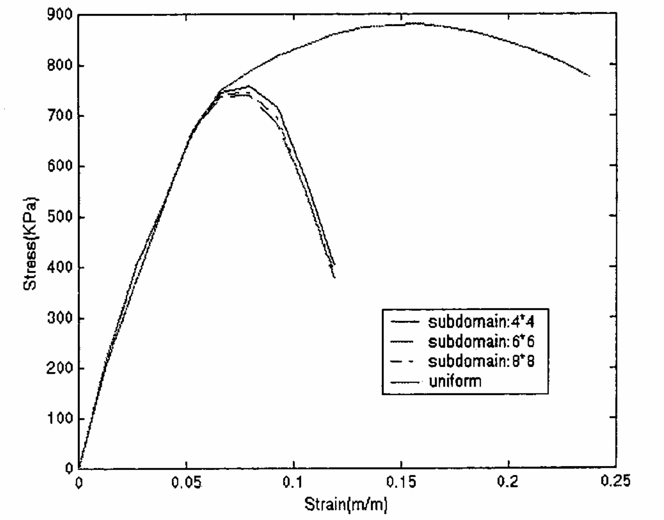
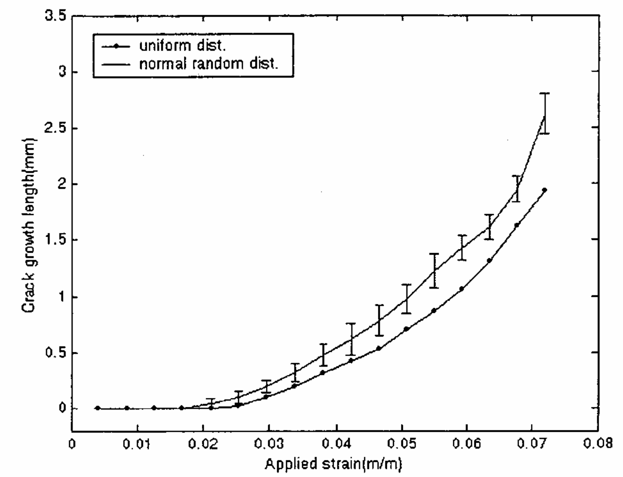
Inhomogeneity of Micro-structures Has a Significant Effect on the Failure Property and Crack Growth Behavior in Solid Propellants

- A meso-macro multi-scale analysis was used to model the constitutive and the crack growth behavior of a solid propellant.
- The Young's modulus is a micro-structural insensitive material property.
- The failure stress and failure strain as well as the crack growth behavior are highly dependent on the material's micro-structure.





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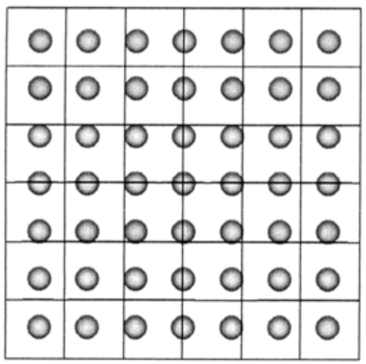
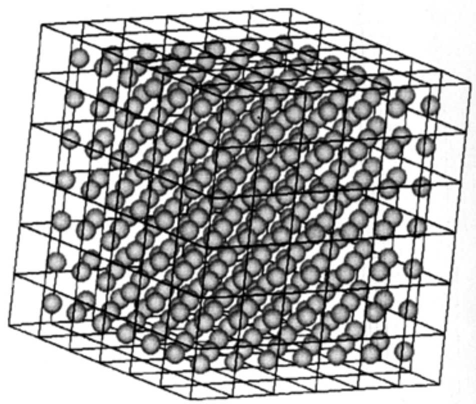
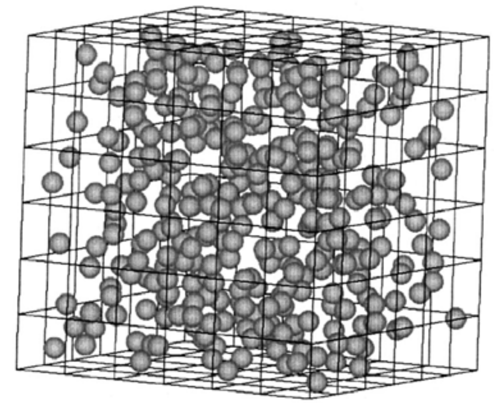
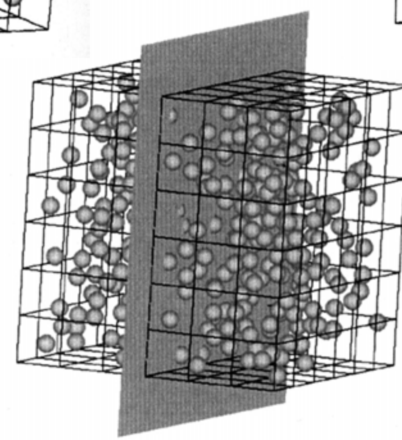
For the 6 micron AL Particles, particle arrangements within a unit cell have no effect on the effective Young's modulus and the effective Poisson's ratio of the particulate composite material



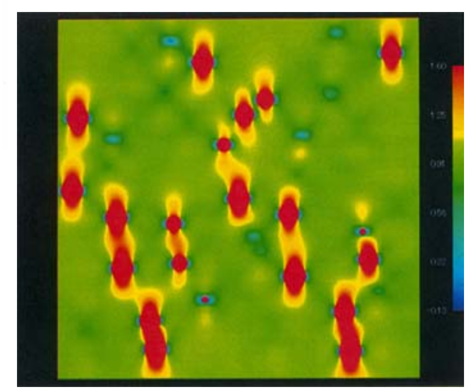




Particle Arrangement	***Effective Young's Modulus***	***Effective Poisson's Ratio***
Random	**0.51 MPa**	**0.4999**
Regular	**0.51 MPa**	**0.4999**



Particle Size	***Young's Modulus (Test)***
6 micron	**0.53 MPa**
0.2 micron	**0.66 MPa**



Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials

Conclusions:

1) The tensile strength and the Young's modulus of TPEG reinforced with 0.2 micron AL particles are higher than those of TPEG and TPEG reinforced with 6 micron AL particles.
2) The local deformation mechanisms (large displacement and void formation) near the crack tip for the three materials studied are similar but the damage mechanisms for the pure binder and the particle reinforced binder are different.
3) The interfacial strength between the AL particle and the binder is weak.
4) Based on the multi-scale modeling analysis, the Young's modulus is independent of material's microstructure. However, the tensile strength is highly dependent on the microstructure.
5) Based on the unit cell model, particle arrangements (regular and random) have no effect on the predicted effective Young's modulus and Poisson's ratio.
6) The degree of inhomogeneity of material's micro-structure and the number of non-propagating cracks increase as the applied strain is increased.
7) A good correlation exists between the dilatations measured by X-ray technique and dilatometer.

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